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LLNL's Experience with the 3rd ITWG Nuclear Forensics Round Robin - INMM 2011

M. J. Kristo

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LLNL's Experience with the 3rd ITWG Nuclear Forensics Round Robin

Michael J. Kristo
Lawrence Livermore National Laboratory
7000 East Avenue
Livermore, CA 94550

ABSTRACT

The Nuclear Forensics International Technical Working Group (ITWG) is an organization of technical experts, law enforcement officials, policy makers, and diplomats from interested governments who cooperate to identify best practices in the field of nuclear forensics. The ITWG was established some fifteen years ago to advance the science of nuclear forensics by exchanging information, developing procedures and recommendations, and conducting international exercises. In fact, conducting practical exercises has been one of the organization's key activities from the beginning. In 1999, ITWG conducted an exercise involving the analysis of plutonium oxide powder from the European civil nuclear cycle. In 2003, ITWG conducted an exercise involving the analysis of HEU oxide powder. This past year, ITWG conducted an exercise involving paired samples of HEU metal. LLNL is one of only three laboratories to have participated in all three ITWG exercises. All participants were required to analyze the provided materials to the limits of their self-declared capability. In addition, participants were requested to analyze the materials in accordance with the ITWG Model Action Plan (see IAEA Nuclear Security Series No. 2), which provides for 24-hour, 1-week, and 2-month reporting. Since the two previous exercises, and, in fact, most real interdicted samples, had involved oxide powders, the analysis of intact metal pins provided both challenges and opportunities to the implementation of the Model Action Plan. For example, it became important to take the time to fully analyze and document the intact specimens before any sub-sampling took place. One of the key requirements for the exercise was the comparison of the two samples. Participants were requested to determine how the two samples might be related to each other. In doing so, we found that the two samples, although quite similar in many ways, suggesting a similar origin, had key differences which definitively proved that they were not from the same batch of material. We also found that bulk age dating can be problematic for uranium metal of complex history. Our analysis of multiple radiochronometers became important for unraveling the complex history of the material, thus allowing correct interpretation of the age dating results.

THE NUCLEAR FORENSICS INTERNATIONAL TECHNICAL WORKING GROUP

The Nuclear Forensics International Technical Working Group (ITWG) is an organization of technical experts, law enforcement officials, policy makers, and diplomats from interested governments who cooperate to identify best practices in the field of nuclear forensics. The first organizational meeting of the ITWG was held at LLNL in 1995 with the goal of advancing the science of nuclear forensics by exchanging information, developing procedures and

recommendations, and conducting international exercises.¹ Sixteen additional meetings of the ITWG have been held since that time, with the number of participating countries increasing from 14 to 30, and the number of participants reaching 80 at the recent meeting of the ITWG in Kiev, the Ukraine.

Conducting practical exercises has been one of the organization's key activities from the beginning. In 1999, ITWG conducted an exercise involving the analysis of plutonium oxide powder from the European civil nuclear cycle. Six international laboratories, including LLNL, participated in this round robin. In 2003, ITWG conducted an exercise involving the analysis of HEU oxide powder. Ten international laboratories, including LLNL, participated in this round robin.^{2,3} This past year, ITWG conducted an exercise involving paired samples of HEU metal. Nine international laboratories, including LLNL, participated in this round robin.⁴

THE THIRD ITWG ROUND ROBIN

The samples for the third ITWG round robin were shipped to the participants in February 2010. All information about the source and nature of the samples, except for that information necessary for proper receipt and materials accounting, were withheld from the participants until the final Data Review Meeting held on September 15-17, 2010, in Dijon, France.

Two 161-type storage castings of HEU metal at Y12 were chosen to be the source of the exercise material. 161-type storage castings are annular in shape with the following dimensions: 12.700 cm O.D., 8.890 cm I.D., and heights ranging from 14.559 to 14.856 cm.⁵ A ring of approximately 3 mm in thickness was cut from the center of each casting using a band saw. A series of trapezoidal pins was then cut from each of the two rings by making quick radial cuts with a band saw. The resulting pins were 5-6 grams in mass, with approximate dimensions of 18 mm in length, 3 mm in thickness, and 3-5 mm in width at either end. Each laboratory received 2 pins, one from each of the two original castings.

The participating laboratories were requested to analyze the samples in accordance with their self-declared capabilities, as well as the ITWG Model Action Plan,^{6,7} including 24-hour, 1-week, and 2-

¹ D. K. Smith, T. Biro, B. Chartier, K. Mayer, S. Niemeyer, and P. Thompson, "Recent Activities of the Nuclear Smuggling International Technical Working Group to Thwart Illicit Trafficking," Proceedings of the IAEA Conference on Illicit Nuclear Trafficking, Edinburgh, Scotland, November 19-22, 2007, pp. 389-396.

² G. Dudder, R. Hanlen, G. Herbillon, "International Technical Working Group Round Robin Tests," from the Proceedings of the IAEA Conference on Advances in Destructive and Non-destructive Analysis for Environmental Monitoring and Nuclear Forensics, held at Karlsruhe, Germany, October 21-23, 2002, pp. 41-51.

³ G. Dudder, R. Hanlen, G. Herbillon, "ITWG Round Robin Tests," presented at the 230th National Meeting of the American Chemical Society, held at Washington, DC, August 28-September 1, 2005, pp.U2305-U2306.

⁴ R. Hanlen, "The Importance of International Nuclear Forensics Analytical Exercises," presented at the Global Initiative to Combat Nuclear Terrorism's workshop on Nuclear Forensics and Legal Aspects of Combating Nuclear and Radiological Terrorism, held at Jerusalem, Israel, June 7-9, 2010.

⁵ S. McConchie, P. Hausladen, and J. Mihalczko, "Passive Time Coincidence Measurements with Assemblies of HEU and DU Castings," Proceedings of the 50th Annual Meeting of the Institute for Nuclear Materials Management, 2009.

⁶ D. K. Smith, M. J. Kristo, S. Niemeyer, and G. B. Dudder, "Documentation of a model action plan to deter illicit nuclear trafficking," J. Radioanalytical & Nuclear Chem., 276 (2008), 415.

month reporting of results. Exercise coordinators also provided a fictional scenario for the exercise. Laboratories were required to determine whether the laws of the fictional country in question, regulating transport of more than 1 gram of uranium enriched to more than 1% ^{235}U , had been violated and whether the two samples (ostensibly seized in separate interdictions) were related.

SAMPLE RECEIPT

The DOT 7A/Type A drum from Y12 was received by the Nuclear Forensics Team at LLNL on February 23, 2010. The drum was opened, and the real-time clock for the exercise commenced on the morning of February 26. Inside a series of nested containers and material packaging, we removed two snap-lock plastic bottles with the samples clearly visible inside. We initiated chain-of-custody on container 3C19VLL6D7 as 10-1-1 and container 3C19VLLVDJ as 10-1-2. We subsequently learned that 10-1-1 was Sample B in the exercise and 10-1-2 was Sample A. Both sets of identifiers were used in our reporting.

In our initial examination, we noted that both samples 10-1-1 and 10-1-2 were solid, pin-shaped samples, nominally 3 mm in thickness and 18 mm long. They were roughly trapezoidal in cross-section. Sample B (10-01-01) was approximately 6 mm at one end and 4 mm at the other end of the trapezoid, while Sample A (10-01-02) was approximately 5 mm at one end and 3 mm at the other end of the trapezoid. All surfaces appeared to have a slightly oxidized surface and otherwise appeared to be unfinished. In addition to standard photo documentation, we also took initial photomicrographs for each sample. During this evaluation, we noticed that each sample had the number “16” or “91” written by hand with what appeared to be a felt-tipped marker on both of their long, thin sides (~3 mm x 18 mm). The number was sometimes aligned with the long dimension of the piece, sometimes aligned perpendicular to the long dimension of the piece.

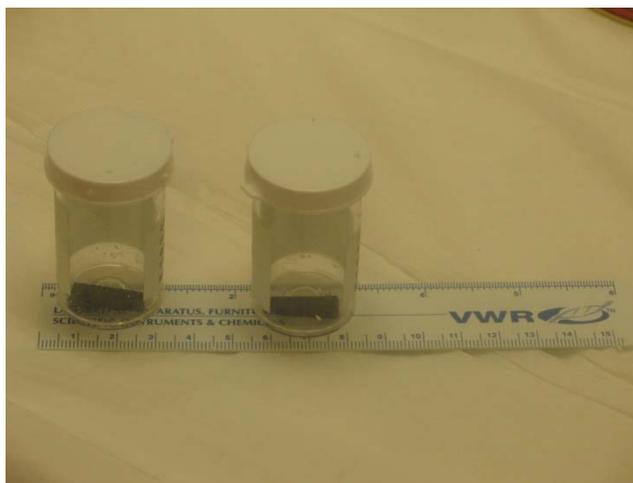


Figure 1. Two sample bottles.
Left: Sample B (10-1-1) Right: Sample A (10-1-2)

Also during our initial examination, we measured the mass of 10-1-1 as 5.6196 ± 0.0002 grams and the mass of 10-1-2 as 5.0640 ± 0.0002 .

⁷ “Nuclear Forensics Support,” IAEA Nuclear Security Series No. 2, International Atomic Energy Agency, Vienna, 2006.

We subsequently undertook a more rigorous measurement of the sample dimensions (see Figures 2 and 3). Assuming that the cuts were made perfectly along the radius of the ring, we estimated the I.D. for each piece as ~ 8.1 cm for 10-1-1 and ~6.4 cm for 10-1-2. The calculation for 10-1-1 is close to the true dimensions of the storage casting, while that for 10-1-2 is not, showing that the cuts were not perfectly radial.

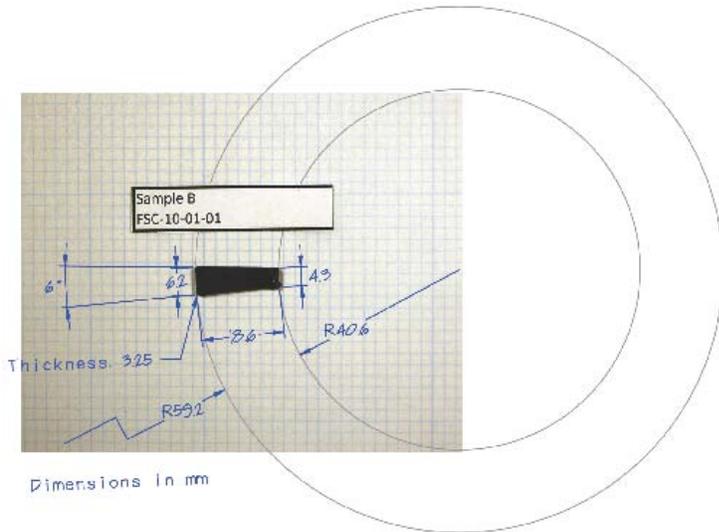


Figure 2. Dimensions of 10-1-1

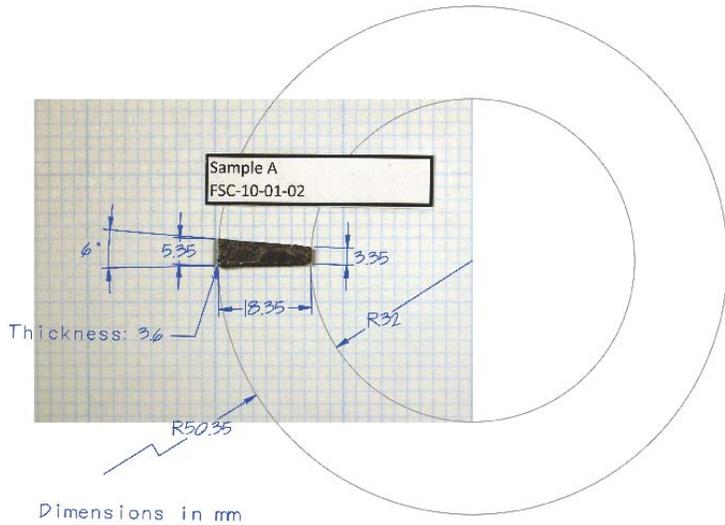


Figure 3. Dimensions of 10-1-2

INITIAL NON-DESTRUCTIVE ANALYSIS

We performed visible-near infrared reflectance spectroscopy on the samples and collected samples of associated organic compounds for gas chromatography-mass spectrometry analysis using semi-permeable membrane extraction (SPME). In this case, neither technique could distinguish between the two samples, nor provide unique insight into the source or production history of the material.

We then performed gamma spectrometry on the intact samples for initial categorization only. Higher accuracy and precision were obtained from whole solution gamma spectrometry and inductively coupled plasma/mass spectrometry performed later. This initial gamma spectrometry measurement indicated that the samples were HEU (both $94 \pm 2\%$ ^{235}U). This measurement, combined with the mass measurement above, gave the first indication that the laws of the fictional country in the scenario had been violated. In addition, we detected ^{232}U and ^{237}Np , indicating that the feed for the enrichment process that produced the HEU in question must have included some reprocessed reactor fuel. After 24-hours, we were unable to find any distinguishing characteristics between the two samples (other than slight differences in dimensions).

After whole sample gamma spectrometry, the exterior of samples A and B were fully documented by optical microscopy. The exterior of both samples were quite similar, although Sample A (FSC 10-1-2) appeared to have a greater number of surface “decorations” (obvious features, perhaps inclusions or occlusions, of different composition than the bulk material). For both samples, the dominant feature of the two large flat surfaces was a series fairly regular grooves or striations ($\sim 15/\text{mm}$), which turned out to be caused by the band saw. The long, narrow sides of both pieces also showed striations, but more irregular and widely spaced. Both ends of each piece had a uniform, oxidized appearance, indicating that these surfaces were as-cast. We again noted the number “16” or “91” (see Figure 5).



Figure 4. Flat side of FSC 10-1-1

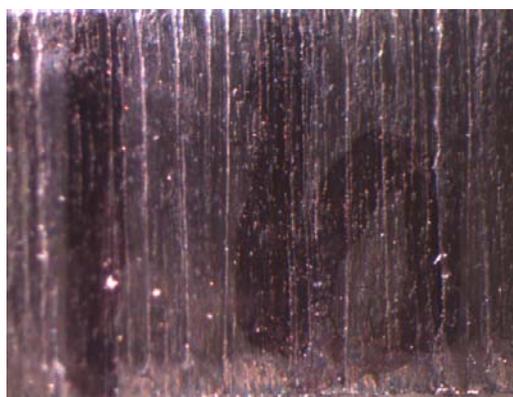


Figure 5. Edge of FSC 10-1-2

Since several “interesting” features were found during optical microscopy, SEM was used to investigate the nature of these observed features. Once in the SEM, though, it was agreed that a more thorough effort to document all 6 surfaces for each of the two samples was prudent. Images were taken with overlap at 100x for each of the six surfaces using the secondary electron detector on an FEI INSPECT- F FE-SEM (15keV accelerating potential). On occasion, images were also taken with the backscatter detector to document the compositional variation of the materials observed on the surface. We collected higher resolution images and EDS spectra from at least one (often three) area of interest on each side.

In general, evidence for mechanical markings is clearly visible on all four of the long sides of the samples with no evidence on the ends of similar markings. Several impurities were identified both in, and on, the metal pin, with compositions including various combinations of Fe, Al, C, Ti, Na, and Cl.

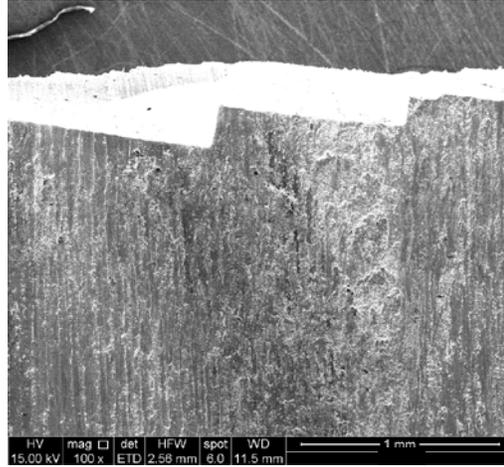
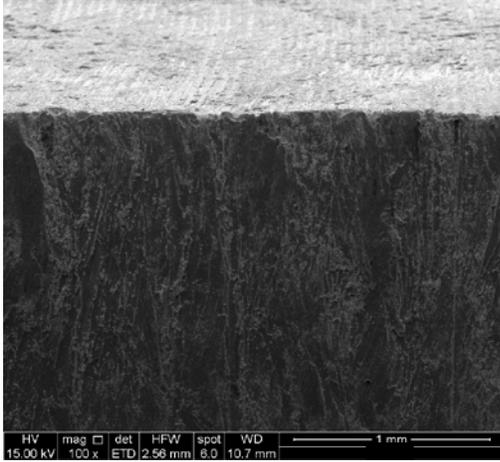


Figure 6. SEM image of edge/flat of 10-1-1

Figure 7. SEM image of rough edge of FSC-10-1-2

SAMPLE ALIQUOTING

Samples were cut into 4 approximately equal-sized pieces on an ISOMET 1000 wet saw. A separate 6-inch diameter diamond wafering blade was used for each sample. As each piece was cut it was placed in a pre-labeled centrifuge tube to air dry. Once all the cuts were completed, each of the centrifuge tubes was filled with approximately 4ml of acetone, shaken, and the acetone and sample were poured out onto a cotton swipe. Weights were then taken and recorded after the acetone had dried (as determined by weight stability). Both saw and process blanks were prepared and processed in parallel with the samples

ISOTOPE RATIO MASS SPECTROMETRY

The sub-samples for 10-1-1 and 10-1-2 were dissolved, relevant elemental fractions were separated and purified, and the resulting solutions analyzed by multicollector inductively coupled plasma-mass spectrometry (MC-ICP-MS) using a NuPlasma HR. Uranium isotopic results are listed in Table 1. Uranium assays were also determined: 99.55 (± 0.25) % U for 10-1-1 and 99.57 (± 0.25) % U for 10-1-2. Plutonium isotopic results are listed in Table 2. Neptunium concentrations were determined as 3.42 \pm 0.12 ppm for 10-1-1 and 4.41 \pm 0.15 ppm for 10-1-2.

Table 1. Uranium isotopic composition from MC-ICP-MS

Sample ID	Atom Percent				
	233U	234U	235U	236U	238U
FSC-10-1-1	0.0000431(44)	0.97768(41)	91.5078(94)	0.40618(59)	7.1083(77)
FSC-10-1-2	0.0000329(44)	1.00370(40)	92.9832(85)	0.38597(56)	5.6271(61)

Table 2. Plutonium isotopic composition from MC-ICP-MS

Sample ID	atomic ratios		
	240Pu/239Pu	241Pu/239Pu	242Pu/239Pu
10-1-1	0.06238(43)	0.000556(11)	0.001086(25)
10-1-2	0.06574(45)	0.000662(13)	0.001375(38)
Analysis date for the Pu isotopes is 18-Mar-10			

TRACE ELEMENTAL IMPURITIES

Aliquots of the initial dissolutions were analyzed by quadrupole inductively coupled plasma-mass spectrometry using a Thermo X-7 ICP-MS for trace impurity concentrations. 15 elements were detected above background in both samples. Many of them were common impurity elements, such as Fe, Cr, and Mn. However, we also detected Zr and Er, both of which can be used in the oxide form as mold coatings for uranium casting. However, no Y, also a potential mold coating, was detected in either sample. We also detected Mo, W, Re, and Ir, all of which form volatile fluorides and could potentially survive the enrichment process from impurities in the feed.

Some of the fines from the cutting (aliquoting) operation were analyzed by stable isotope mass spectrometry for C, N, O, and S content, as well as C isotopic composition. No N or S was detected. We did detect O above background in 10-1-1, but this is presumed to be from surface oxidation of the HEU. The carbon content of both samples was extremely high ($0.115 \pm 0.007\%$ in 10-1-1 and $0.170 \pm 0.005\%$ in 10-1-2), consistent with heavily recycled uranium material. The $\delta^{13}\text{C}$ values (-21.3 per mil for 10-1-1 and -21.2 per mil for 10-1-2) were consistent with C3 plants, coal or graphite. The fact the measured values are the same within analytical uncertainty, strongly suggesting that the carbon came from the same source in both samples.

RADIOCHEMISTRY/NUCLEAR COUNTING

Sub-samples from 10-1-1 and 10-1-2 were dissolved, relevant elemental fractions were separated and purified, and the resulting solutions analyzed by alpha spectrometry. We rely on radiochemistry/counting to measure ^{232}U and ^{238}Pu (see Table 3).

Table 3. Isotopic measurements uniquely provided by RC/Alpha spectrometry

Sample ID	atomic ratios	
	$^{232}\text{U}/^{235}\text{U}$	$^{238}\text{Pu}/^{238}\text{Pu}$
10-1-1	$1.45(\pm 0.21)\text{E-}10$	$2.390(\pm 0.093)\text{E-}3$
10-1-2	$1.15(\pm 0.19)\text{E-}10$	$4.657(\pm 0.093)\text{E-}3$

SEM/EDS/EMPA CHARACTERIZATION OF POLISHED SURFACES

Samples were cut for metallurgical analysis along orthogonal axes, embedded in epoxy and polished using carborundum-impregnated discs. Final polishing was performed with 3- and 1-

micron diamond paste. Samples were examined in a JEOL JSM-7401F SEM equipped with an Oxford Inca X-max 80 EDS (10 keV). Quantitative analyses of uranium carbides were performed with a JEOL JXA-8200 electron microprobe.

Both samples contain similar populations of small inclusions. The different types of inclusions exhibit different characteristic morphologies, making it possible, in many cases, to identify an inclusion based solely on appearance. Uranium carbide (U-C) is the most abundant, followed by uranium phosphide (U-P-C), uranium boron-carbide (U-B-C), Fe-Ni-U carbide and (possibly) SiC. Since carborundum was used to prepare the samples, we cannot exclude the possibility that some of the SiC is a contaminant. Quantifying the chemical composition of the inclusions based on SEM/EDS analyses was not possible due to the intense x-ray emission corresponding to the U α transition. However, the results from quantitative analyses of 10 larger U-C inclusions using wavelength dispersive analysis on the electron microprobe indicated that the larger, orthorhombic U-C inclusions are uranium monocarbide (UC).

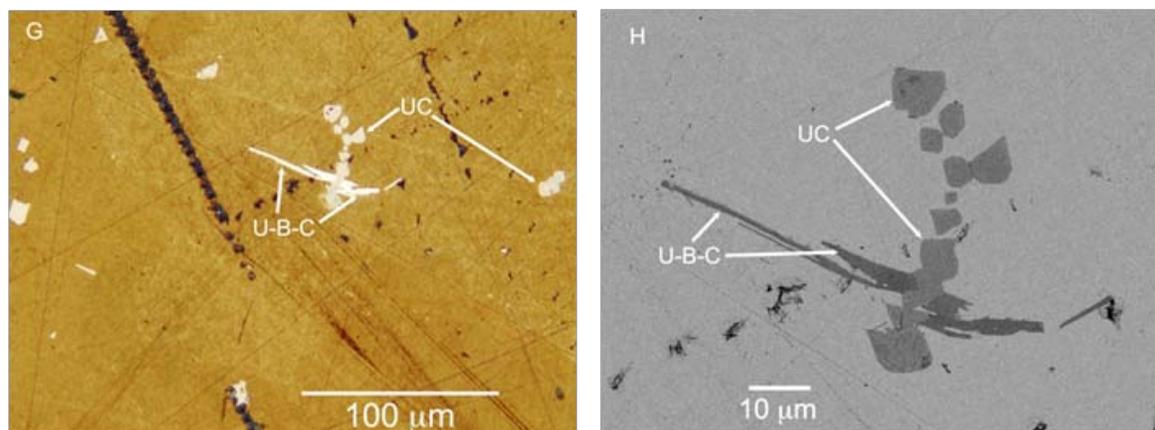


Figure 8. Paired optical (lefthand column) and SEM backscattered electron (righthand column) photomicrographs of inclusions in the U-metal samples.

AGE DATING

Age dating, the determination of time since chemical purification, is a commonly used technique in nuclear forensics.^{8,9,10} Accurate age dating relies on several assumptions. First of all, there must be a purification event that quantitatively removes daughter (product) nuclide from the parent (precursor) nuclide. Incomplete purification leads to calculated ages (model ages) that are older than the true time since purification. Second, the sample must remain a closed system from purification until sampling and analysis, precluding both either contamination from outside the system and segregation of either daughter or parent. We calculated “model ages for both samples using several isotopic systems: $^{234}\text{U} \rightarrow ^{230}\text{Th}$, $^{241}\text{Pu} \rightarrow ^{241}\text{Am}$, and $^{235}\text{U} \rightarrow ^{231}\text{Pa}$ as shown in Table 4.

⁸ S. Lamont and G. Hall, “Uranium age determination by measuring the $^{230}\text{Th}/^{234}\text{U}$ ratio,” *Journal of Radioanalytical and Nuclear Chemistry*, Vol. 264, No. 2 (2005) 423-427.

⁹ Wallenius, M., Mayer, K., “Age determination of plutonium material in nuclear forensics by thermal ionisation mass spectrometry”, *Fresenius’ J. Anal. Chem.* Vol. 366, No. 3, 2000, 234-238.

¹⁰ A. Morgenstern, C. Apostolidis, and K. Mayer, “Age Determination of Highly Enriched Uranium: Separation and Analysis of ^{231}Pa ,” *Anal. Chem.* 2002, 74, 5513-5516.

When possible, model ages calculated from radiochemistry and alpha spectrometry are compared with those calculated from mass spectrometry.

Table 4. Model ages calculated from both mass spectrometry and alpha spectrometry.

			Mass Spec	Alpha Spec	Years since casting
Measurement	Sample	Units	Value		
234U-230Th Age	B	years	6.041(36)	5.62(65)	6.10
	A		6.861(41)	7.02(67)	6.82
235U-231Pa Age	B	years	35.77(37)		
	A		33.73(36)		
241Pu-241Am Age	B		17.46(37)	15.3±1.9	
	A	years	14.35(33)	12.3±1.3	

There are two striking features in these results. First, we note the correspondence between the model age, calculated from the ^{234}U - ^{230}Th system, and the time since casting (revealed only during the Data Review Meeting). Second, we note the lack of agreement between any of the 3 model ages. Clearly, the casting process caused a segregation of the Th in the material, such that Th was completely removed from the center of the casting (where the round robin samples were taken). However, this process did not result in the complete removal of other impurities, such as Pu, Am, or Pa. The segregation of radioactive impurities into the top of uranium castings (the so-called “hot top”) has been observed for some time. Higher concentrations of C in the U have been associated with greater removal of some species, due to a self-slagging process.^{11,12,13,14} It appears that Th has been completely removed from the center of the casting by this self-slagging process, while the other species have been removed partially or not at all.

Without extrinsic knowledge of the sample, we have to be very careful in interpreting model ages. For instance, we did not know *a priori* anything about the casting shape (other than what we deduced from the pin shape) or sampling process. The pin could have been taken from the “hot top,” for example, and resulted in the determination of an anomalously old model age. Even now, with knowledge of the casting shape and process, we still don’t know how generalizable this result is. We still lack the fundamental studies necessary to extrapolate this single point measurement to the more general case where the conditions of casting might be expected to vary widely (with unknown effects on impurity segregation).

The measurement of multiple chronometers in nuclear forensics is critical for testing whether the fundamental assumptions of age dating have held true for the sample in question. If we obtain agreement between model ages from different systems, we can posit that age as the true time since purification more confidently, since the likelihood that two or more chemical systems will be contaminated or fractionate in precisely the same way is very low. On the other hand, if we obtain disagreement between model ages from multiple systems, then we know to exercise caution when interpreting those ages. In fact, such systematic

¹¹ F. Martin and G. Miles, “The Processing of Irradiated Uranium by High Temperature Oxidation Reactions,” in *Process Chemistry*, Series III of Progress in Nuclear Energy, ed. By F. Bruce, J. Fletcher, H. Hyman, and J. Katz, pp. 329-341.

¹² H. Feder, N. Chellew, and M. Ader, “Melt Refining of Uranium,” in *Process Chemistry*, Series III of Progress in Nuclear Energy, ed. By F. Bruce, J. Fletcher, H. Hyman, and J. Katz, pp. 301-308.

¹³ J. Antill, E. Barnes, and M. Gardner, “Zone Melting of Uranium,” in *Progress in Nuclear Energy* (1959), pp.9-18.

¹⁴ C. Whitman, V. Compton, R. Holden, “Zone Melting of Uranium,” *J. Electrochem. Soc.*, 104, No. 4 (1957), pp. 240-244.

variations in the ages can often provide insight into the chemical and physical processes used in preparing the material.¹⁵

TECHNICAL INTERPRETATION

From the preceding results, we were able to make the following general conclusions. All uranium isotopic analyses indicate that both samples are weapons-usable, highly enriched uranium. Therefore, both seizures indicate that the statutes of the fictional country in the exercise, regarding the transport of uranium materials have been violated (>1 gram, >1% enriched in U-235). The two samples have isotopic compositions and model ages that differ well outside analytical uncertainty ($k=2$, indicating that, even with all of the other similarities between the two samples, the two questioned samples originate from different source materials).

The enrichment levels for both samples were similar to, but different than, 93% enrichment values found in several countries, e.g., the U.S. On a 3-isotope plot ($^{238}\text{U}/^{235}\text{U}$ ratio versus $^{234}\text{U}/^{235}\text{U}$ ratio), ITWG Sample A and Sample B lie along a mixing line ($R^2=0.9998$) between US 93% and 70% enriched. NBL U650 (65% U-235) and U750 (75% U-235), also produced from material from the US diffusion enrichment system, lay well off (and on opposite sides of) this mixing line. Of course, U.S. production had some internal process variability, but not enough to obscure 70% and 93% as likely end members for this mixing line.

The $^{234}\text{U}/^{235}\text{U}$ ratio indicates likely isotopic enrichment via either a gaseous diffusion or centrifuge process and not via electromagnetic or laser isotope separation. The presence of ^{232}U , ^{233}U , ^{236}U , and ^{237}Np indicates that some of the enrichment feed stock had been irradiated in a reactor. The presence of trace levels of weapons-grade Pu further indicates an origin from a country with weapons based upon both fissile materials.

The geometry of these samples suggests that they are pieces from a larger part of annular cross-section, a typical storage configuration for HEU, implying that the original material may have been part of a location that stores significant amounts of HEU. Uranium parts are typically cast using graphite molds with coatings of erbia, zirconia, or yttria. The high level of carbon in these samples likely originates from the graphite molds –the molds used for this casting, as well as previous castings of the component materials. In fact, the extremely large amount of carbon in these samples suggests a significant amount of recycling. We also see residue from the erbia and zirconia mold coatings, which must be from previous, separate castings, since erbia and zirconia are typically not used in combination.

In summary, then, the sum of these observations suggests that these samples are from a large nuclear weapons state. This state probably uses gaseous diffusion for enrichment and includes recycled uranium in the production cycle. Based upon the geometry of the initial object, as extrapolated from the geometry of the interdicted sample, this material was probably diverted from a facility used to storing large amounts of highly enriched uranium. This facility probably also includes the facilities for casting, machining, and cutting uranium parts. This particular material, though, is not highly refined metal and was likely not intended for weapons use without further processing or refinement.

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¹⁵ K. Moody, I. Hutcheon, P. Grant, Nuclear Forensic Analysis (New York: Taylor & Francis, 2005), pp. 237 & 418.